

U. S. FISH AND WILDLIFE SERVICE
PATUXENT ANALYTICAL CONTROL FACILITY
QUALITY ASSURANCE REPORT

NA MANAGER
7 ASSISTANT
____ CLERK
Wt ECOLOGIST *Return*
____ ARCHAEOLOGIST

RE: 5887 REGION: 1 REGIONAL ID: 89-1-100A FILE DESTROY

THE ANALYSES ON THE ABOVE MENTIONED SAMPLES WERE PERFORMED AT:

THE MISSISSIPPI STATE CHEMICAL LABORATORY
BOX CR
MISSISSIPPI STATE, MISSISSIPPI 39762

AFTER A THOROUGH REVIEW OF THE REPORT ISSUED BY THE LABORATORY, I REPORT
THE FOLLOWING OBSERVATIONS AND CONCLUSIONS:

THE ACCURACY, AS MEASURED BY SPIKE RECOVERY, WAS GENERALLY ACCEPTABLE.

THE PRECISION, AS MEASURED BY DUPLICATE SAMPLE ANALYSIS, WAS ACCEPTABLE.

Craig L. Kuke 8-27-90
QUALITY ASSURANCE OFFICER DATE



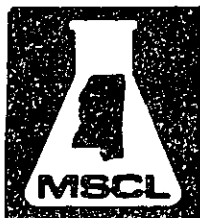
ANALYTICAL REPORT INTEGRITY FORM

Catalog #: 5887 Lab: mscl Region: 1

☒ Initial QA/QC Review -- Report Correct.

[illegible]

MISSISSIPPI STATE UNIVERSITY



MISSISSIPPI
STATE CHEMICAL LABORATORY

BOX CR - MISSISSIPPI STATE, MISSISSIPPI 39762



August 17, 1990

Patty McDonald
Stickel Building/Chemistry
Patuxent Wildlife Research Center
U.S. Fish and Wildlife Service
Route 197
Laurel, MD 20708

Dear Patty:

Enclosed are analytical results for one batch of samples submitted by the U.S. Fish and Wildlife Service (Catalog # 5887, Reg.ID.#: 89-1-100A, Order # 85800-89-08067). The samples were analyzed by Method 6. A Description is enclosed.

Please call if you have any questions.

Sincerely,

Larry G. Lane
Principal Investigator

received
8-21-90

MISSISSIPPI STATE UNIVERSITY
MISSISSIPPI STATE CHEMICAL LABORATORY
BOX CR
MISS. STATE, MS 39762
REPORT FORM
USD1/FWS

Page 1

SAMPLE TYPE: White
Faced Ibis Liver
CAT NO. 5887
Reg. ID.#: 89-1-100A
ORDER NO. 85800-89-
08067

ORGANOCHLORINES

Date P.O. Recd 07/24/89
Date Spis Recd 02/08/90
Queue Date 03/27/90

PARTS PER MILLION AS RECEIVED (WET WT)

FWS #	89-030	89-031	89-032	89-033	89-034	89-035	89-036
LAB #	790258	790259	790260	790261	790262	790263	790264
MATRIX	W.F. Ibis Liver	W.F. Ibis Liver	W.F. Ibis Liver	W.F. Ibis Liver	W.F. Ibis Liver	W.F. Ibis Liver	W.F. Ibis Liver
COMPOUND							
HCB	2.9	0.04	1.5	2.7	0.02	1.6	1.5#
α -BHC	ND*	ND	ND	ND	ND	ND	ND
γ -BHC	ND	ND	ND	ND	ND	ND	ND
β -BHC	ND	ND	ND	ND	ND	ND	ND
δ -BHC	ND	ND	ND	ND	ND	ND	ND
Oxychlordane	ND	ND	ND	ND	ND	ND	ND
Hept. Epox.	ND	ND	ND	ND	ND	ND	ND
γ -Chlordane	ND	ND	ND	ND	ND	ND	ND
t-Nonachlor	ND	0.03	ND	ND	ND	ND	0.03
Toxaphene	ND	ND	ND	ND	ND	ND	ND
PCB's (total)	ND	ND	ND	ND	ND	ND	ND
o, p'-DDE	ND	ND	ND	ND	ND	ND	ND
α -Chlordane	ND	ND	ND	ND	ND	ND	ND
p, p'-DDE	5.6	4.6	4.5	7.9	5.6	8.2	9.6#
Dieldrin	0.05	0.15	0.12	0.08	0.08	0.11	0.17#
o, p'-DDD	ND	ND	ND	ND	ND	ND	ND
Endrin	ND	ND	ND	ND	ND	ND	ND
cis-nonachlor	ND	ND	ND	ND	ND	ND	ND
o, p'-DDT	ND	ND	ND	ND	ND	ND	ND
p, p'-DDD	0.22	0.06	0.20	0.19	0.07	0.13	0.21#
p, p'-DDT	ND	ND	ND	ND	ND	ND	0.04
Mirex	ND	ND	ND	ND	ND	ND	ND
OTHER:							
WEIGHT (g)	-	-	-	-	-	-	-
MOISTURE (%)	70.5	71.5	71.2	72.9	70.0	72.3	72.3
LIPID (%)	4.30	4.90	3.60	ISS	7.20	4.00	4.60

Lower Level of Detection = 0.01 ppm for Tissue, Soil, Etc. 0.05 for Toxaphene and PCBs.
For Water, LLD= 0.005 ppm for OCs, Tox, PCBs
*ND = None Detected **Spike = ppm for
***NS = Not Spiked
= Confirmed by GC/Mass Spectrometry
\$IS = Insufficient Sample

Signature

SAMPLE TYPE: White
Faced Ibis Liver

CAT NO. 5887
Reg. ID #: 89-1-100A
ORDER NO. 85800-89-
08067

ORGANOCHLORINES

Date P.O. Recd 07/24/89
Date Spis Recd 02/08/90
Queue Date 03/27/90

PARTS PER MILLION AS RECEIVED (WET WT)

FWS #	89-037	89-038	Blank	Matrix Blank	Spike**	% Recovery	
LAB #	790265	790266	790267	for	790268		
MATRIX	W.F. Ibis Liver	W.F. Ibis Liver	Reagent	Liver	Liver		
COMPOUND							
HCB	0.04	0.01	ND	ND	0.063	63	
α-BHC	ND*	ND	ND	ND	NS***		
γ-BHC	ND	ND	ND	ND	0.086	86	
β-BHC	ND	ND	ND	ND	0.091	91	
δ-BHC	ND	ND	ND	ND	NS		
Oxychlordane	ND	ND	ND	ND	0.092	92	
Hept. Epox.	ND	ND	ND	ND	0.097	97	
γ-Chlordane	ND	ND	ND	ND	NS		
t-Nonachlor	0.04	ND	ND	ND	0.091	91	
Toxaphene	ND	ND	ND	ND	NS		
PCB's (total)	ND	ND	ND	ND	NS		
o, p'-DDE	ND	ND	ND	ND	0.10	100	
α-Chlordane	ND	ND	ND	ND	0.089	89	
p, p'-DDE	7.3	3.1	ND	ND	0.093	93	
Dieldrin	0.21	0.15	ND	ND	0.091	91	
o, p'-DDD	ND	ND	ND	ND	NS		
Endrin	ND	ND	ND	ND	0.091	91	
cis-nonachlor	ND	ND	ND	ND	0.089	89	
o, p'-DDT	ND	ND	ND	ND	0.097	97	
p, p'-DDD	0.06	0.04	ND	ND	0.10	100	
p, p'-DDT	ND	ND	ND	ND	0.086	86	
Mirex	ND	ND	ND	ND	0.093	93	
OTHER:							
WEIGHT (g)	-	-	-	-	-		
MOISTURE (%)	69.8	71.2	-	75.2	77.0		
LIPID (%)	5.40	3.60	-	4.31	3.80		

Lower Level of Detection = 0.01 ppm for Tissue, Soil, Etc. 0.05 for Toxaphene and PCBs.
For Water, LLD = 0.005 ppm for OCs, Tox, PCBs

*ND = None Detected

**Spike = 0.10 ppm for Liver

***NS = Not Spiked

= Confirmed by GC/Mass Spectrometry

Signature

Larry Lane

Method 6. Analysis For Chlorinated Hydrocarbon Pesticides And Related Compounds – Micro Method

This method is necessary when sample size is limited (below 4 g, approximately) and in case of organ tissue as substrate and is a modified version of the method described in Manual of Analytical Methods for the Analysis of Pesticides in Humans and Enviromental Samples, EPA-600/8-80-038, June 1980, Section 5, A (2). It is suitable for adipose, kidney, liver, muscle, brain, and other tissues:

1. Weigh 0.5 g or less of well-mixed tissue into a size 22 Duall tissue grinder.
2. Extract tissue by grinding three times with acetonitrile; the first time being with 4 ml followed by two 2.5 ml portions.
3. Remove the pestle after each grinding and centrifuge, decanting the extract into a 50 ml glass stoppered graduated mixing cylinder.
4. Combine all extracts and record the total volume of the three extracts.
5. Add a volume of PRQ water equivalent to 3.3 times the extract volume. Then add 2 ml saturated NaCl solution.
6. Extract the aqueous acetonitrile mixture with 5 ml hexane by vigorous shaking for 1 minute.
7. Allow layers to separate, and remove the hexane layer with a Pasteur pipet into a 15 ml screw-capped culture tube.
8. Re-extract twice with 2 ml hexane each time, combining the extracts into the culture tube.
9. Concentrate the combined hexane extracts under nitrogen to approximately 0.5 ml volume.
10. Clean-up on a florisil mini-column as described in Method 2, Steps 8, 9, 10. and 11.

Note For brain tissue additional treatment is necessary before column clean-up:

11. Proceed through Steps 1-9 above, add 0.3 ml acetic anhydride and 0.3 ml pyridine, cap tightly and incubate for 30 minutes in a water bath

Elution Profiles for Florisil, Silica Gel and
Silicic Acid Column Separations

A. Florisil Column:

1. Fraction I (6% ethyl ether containing 2% ethanol, 94% petroleum ether)

HCB, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, oxychlordane, heptachlor epoxide, gamma-chlordane, trans-nonachlor, toxaphene, PCB's, o,p'-DDE, alpha-Chlordane, p,p'-DDE, p,p'-DDT, cis-nonachlor, o,p'-DDT, p,p'-DDD, p,p'-DDT, mirex, dicofol, endosulfan I (Split with FII).

2. Fraction II (15% ethyl ether containing 2% ethanol, 85% petroleum ether)

dieldrin, endrin, dacthal, endosulfan I (split with FI), endosulfan II (split with FIII), endosulfan sulfate (split with FIII).

3. Fraction III (50% ethyl ether containing 2% ethanol, 50% petroleum ether)

endosulfan II (split with FII), endosulfan sulfate (split with FII), malathion.

B. Florisil Mini-Column:

1. Fraction I (12 ml hexane followed by 12 ml 1% methanol in hexane)

HCB, gamma-BHC (25%), alpha-BHC (splits with FII), trans-nonachlor, o,p'-DDE, p,p'-DDE, o,p'-DDD, p,p'-DDD (splits with FII), o,p'-DDT, p,p'-DDT, mirex, cis-nonachlor, cis-chlordane, trans-chlordane, PCB's, Photomirex and derivatives.

2. Fraction II (24 ml 1% methanol in hexane)

gamma BHC (75%), beta-BHC, alpha-BHC (splits with FI), delta-BHC, oxychlordane, heptachlor epoxide, toxaphene, dicofol, dacthal, endosulfan I, endosulfan II, endosulfan sulfate, octachlorostyrene, Kepone (with additional 12mls 1% methanol in hexane).

C. Silica Gel:

1. SG Fraction I (100 ml petroleum ether)

n-dodecane, n-tridecane, n-tetradecane, ocylcyclohexane, n-pentadecane, nonycyclohexane, n-hexadecane, n-heptadecane, pristane, n-octadecane, phytane, n-nonadecane, n-eicosane.

2. SG Fraction II (100 ml 40% methylene chloride in petroleum ether followed by 50 ml methylene chloride)

napthalene, fluorene, phenanthrene, anthracene, fluoranthrene, pyrene, 1,2-benzanthracene, chrysene, benzo [b] fluoranthrene, benzo [k] fluoranthrene, benzo [e] pyrene, benzo [a] pyrene, 1,2:5,6-dibenzanthracene, benzo

[g,h,i] perylene.

D. Silicic Acid:

1. SA Fraction I (20 ml petroleum ether)
HCB, mirex
2. SA Fraction II (100ml petroleum ether)
PCB's, p,p'-DDE (splits with SA III)
3. SA Fraction III (20 ml mixed solvent: 1% acetonitrile, 80% methylene chloride, 19% hexane)
alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, oxychlordane, heptachlor epoxide, gamma-chlordane, trans-chlordane, toxaphene, o,p'-DDE, alpha-chlordane, p,p'-DDE (splits with SAII), o,p'-DDT, cis-nonachlor, o,p'-DDT, p,p'-DDD, p,p'-DDT, dicofol.